Materials Identification and Surveillance – Gas Chromatography Analysis of 3013 Storage Cans 1 and 10

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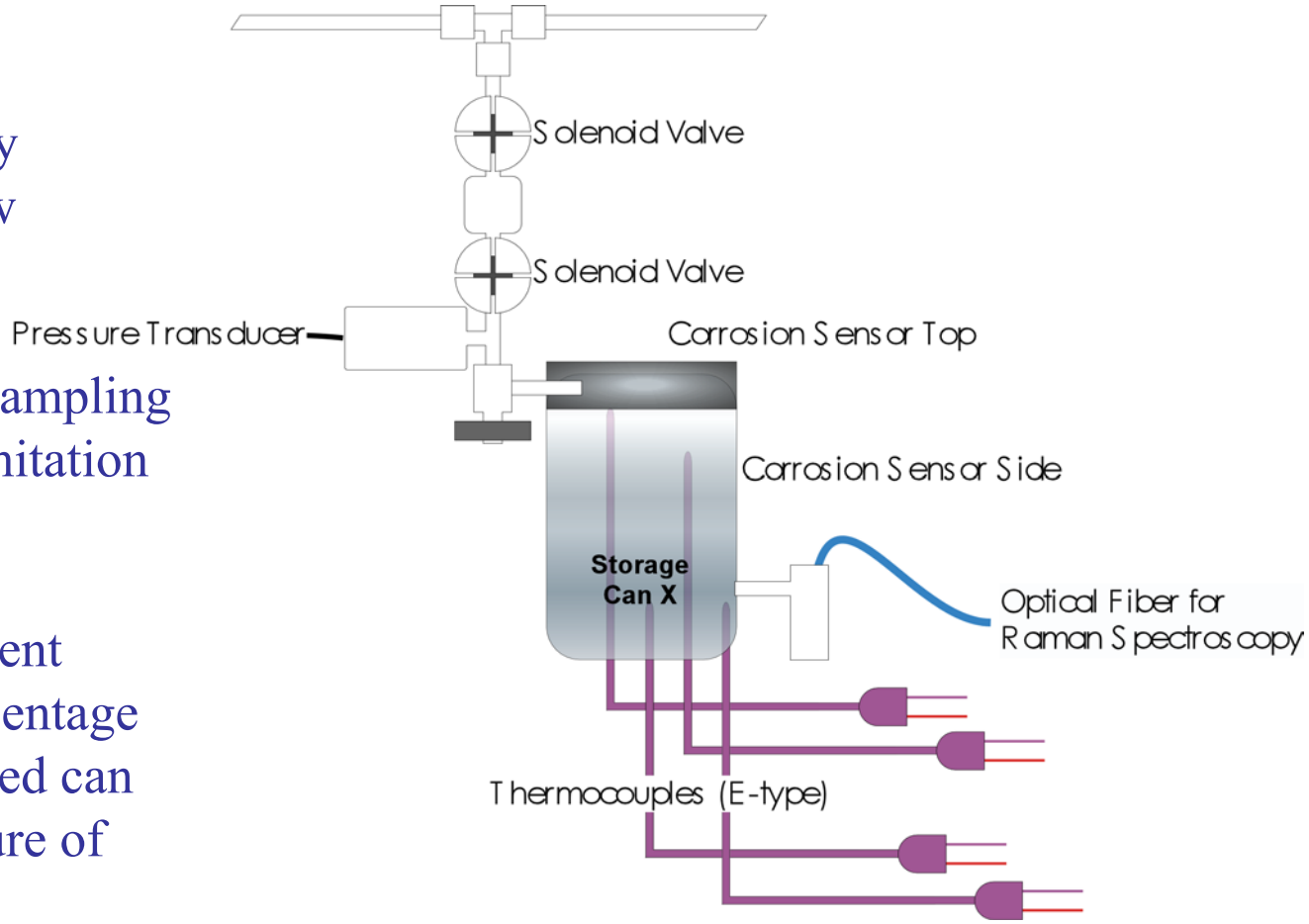
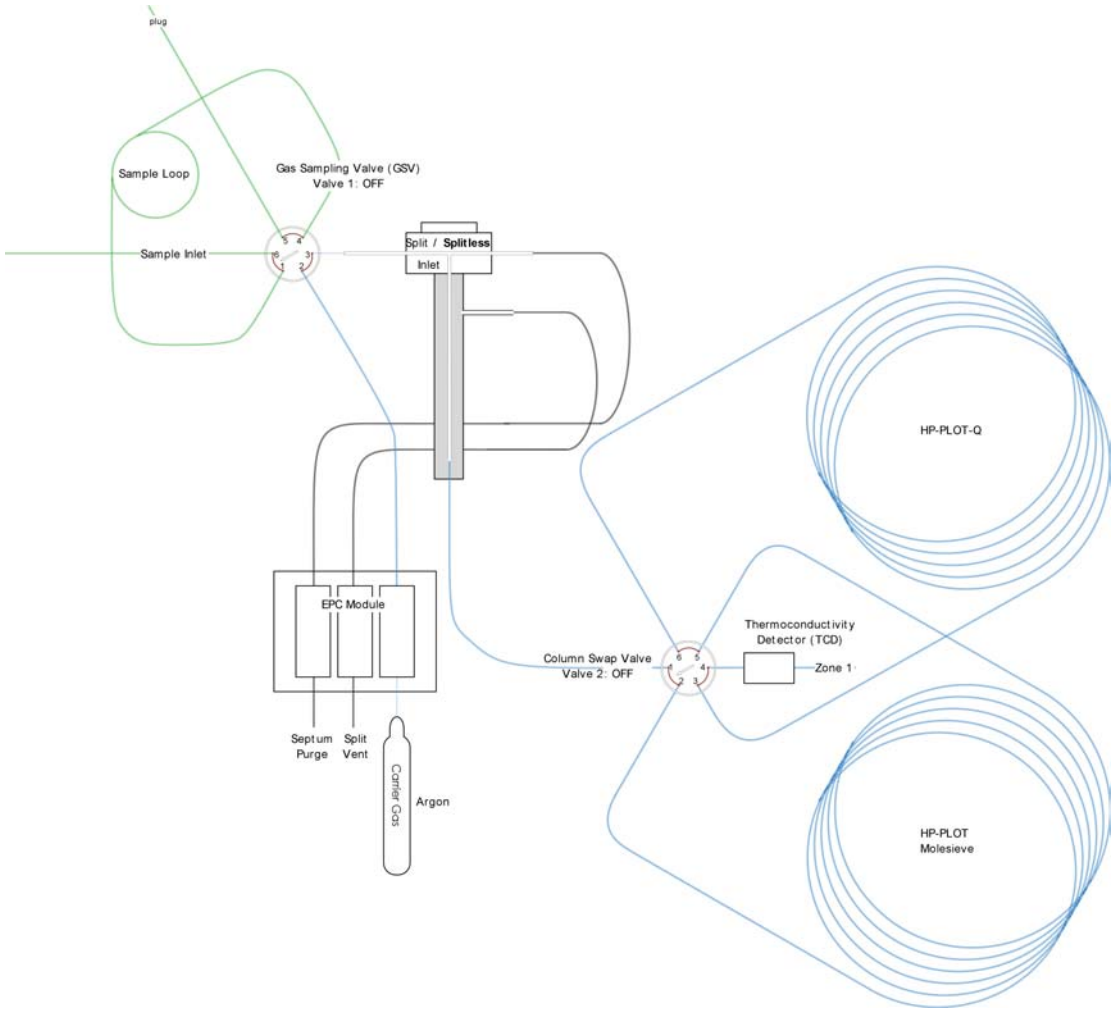
94-1 R&D End of Year Technical Review
October 21 & 22, 2002

GC Sampling Procedures & Sources of Error

- GC sampling completely automated using LabView software.
- Expansion factor from sampling volume to GC ~ 40. A limitation to sensitivity.
- Absolute GC measurement turned into a relative percentage and multiplied by measured can pressure for partial pressure of various gases.



Agilent 6890 Configuration & Sources of Error cont.



- Thermoconductivity (TCD) detector.
- Molsieve & Plot-Q columns in series.
- Argon used as carrier gas for H_2 sensitivity.
- Sensitivity to other gases decreases.
- Peak overlap between He and H_2.

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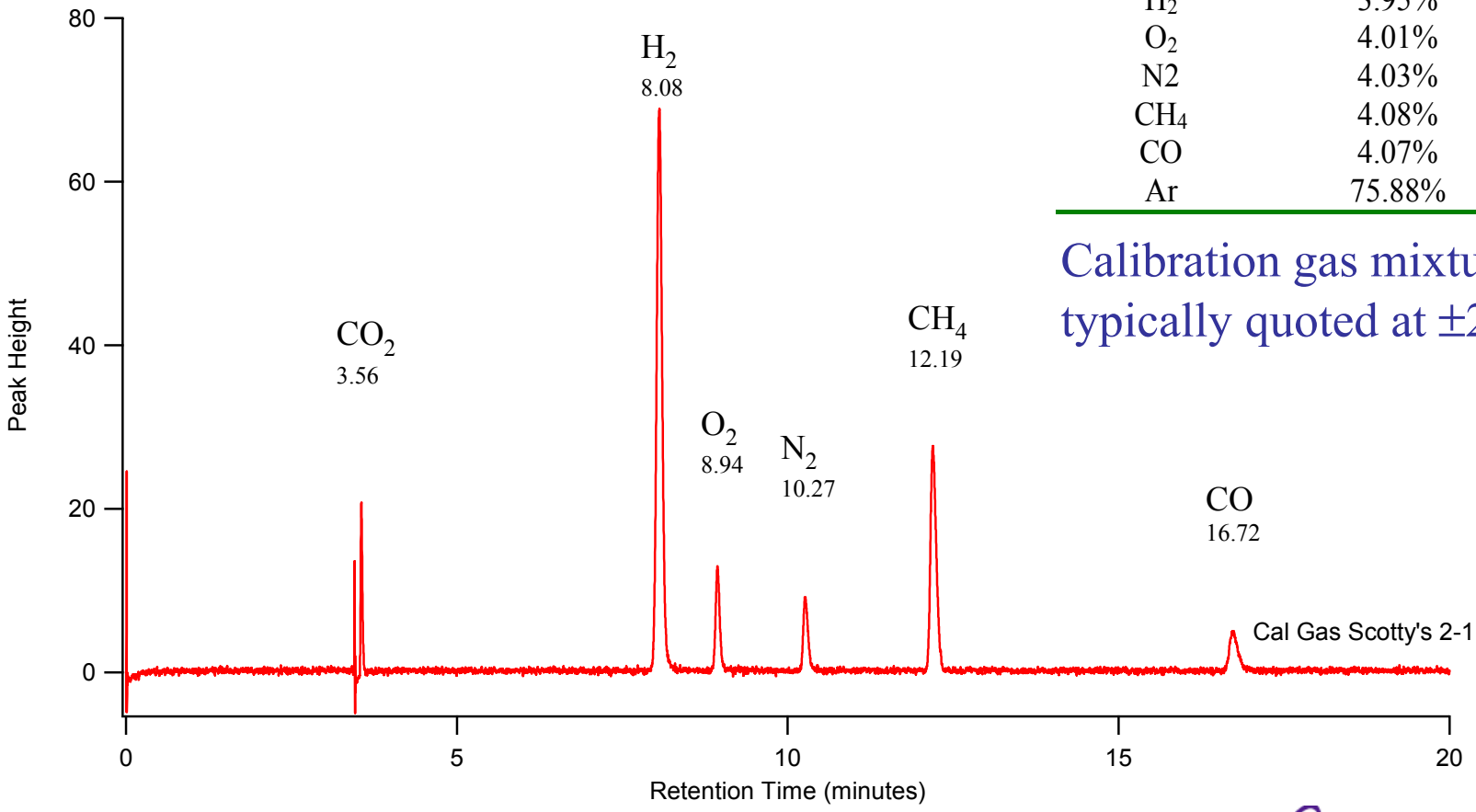
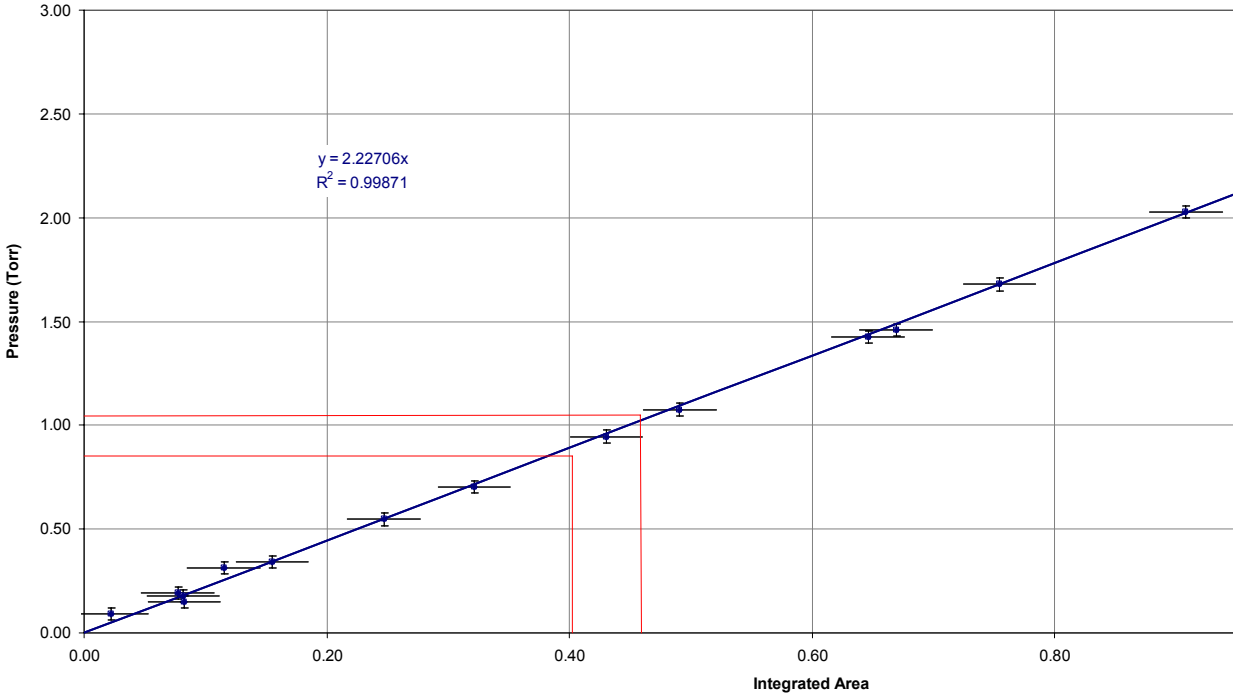
Typical Gas Chromatograph, Agilent 6890

Species	Volume %
CO_2	3.98%
H_2	3.95%
O_2	4.01%
N2	4.03%
CH_4	4.08%
CO	4.07%
Ar	75.88%

Calibration gas mixtures typically quoted at ±2%



Error in Measurement and Detection Limits



Analysis error arises from:

- Measurement error in pressure
- Run to run variations in GC
- Integration errors
- Calibration errors

QA controls:

- 6-12 month multipoint calibration of GC
- single point calibration for each day of use
- Use of "PeakFit for data analysis
- Test of sampling procedure using Can 10

Species	Error (± Torr)	Detection Limit (Torr)
CO_2	3.8	2.0
H_2	1.3	0.5
O_2	2.3	2.0
N_2	2.8	2.0
CH_4	1.2	0.7
CO	4.7	2.0

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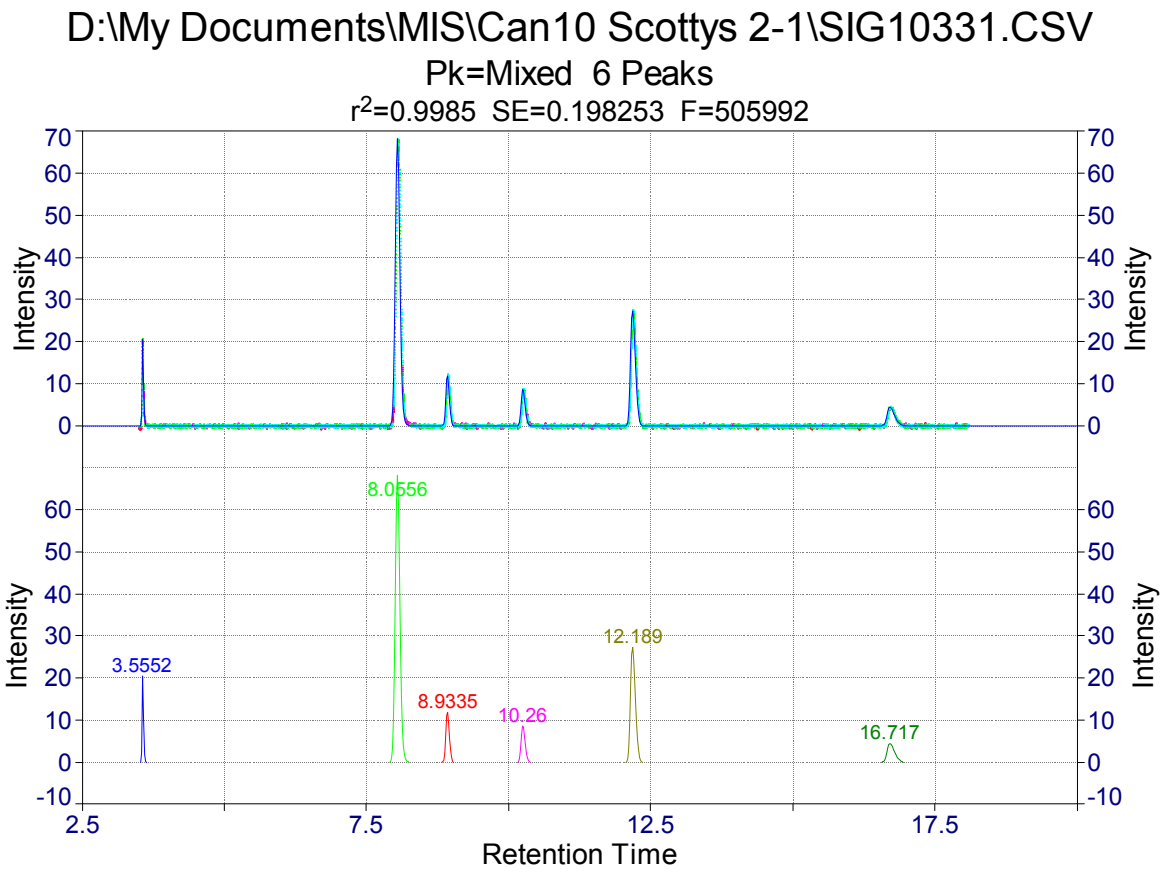
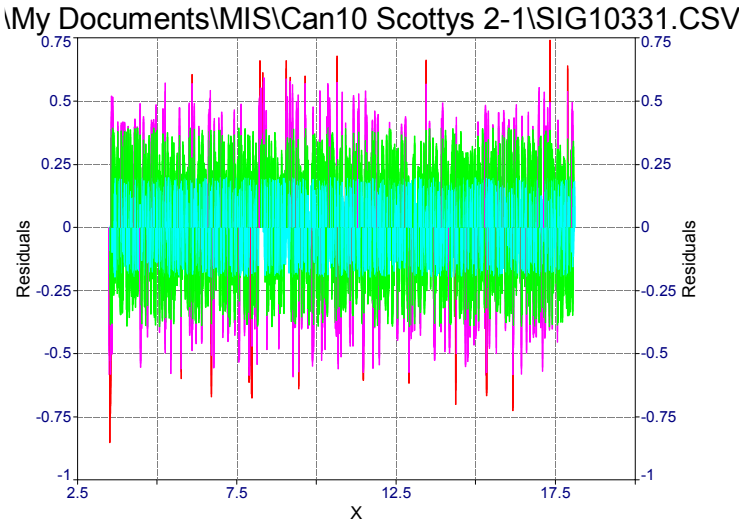


PeakFit Integration

Data analysis involves a multiparameter fit to the baseline followed by curve fitting using either a Haarhoff-Van der Linde or an Exponentially Modified Gaussian function.



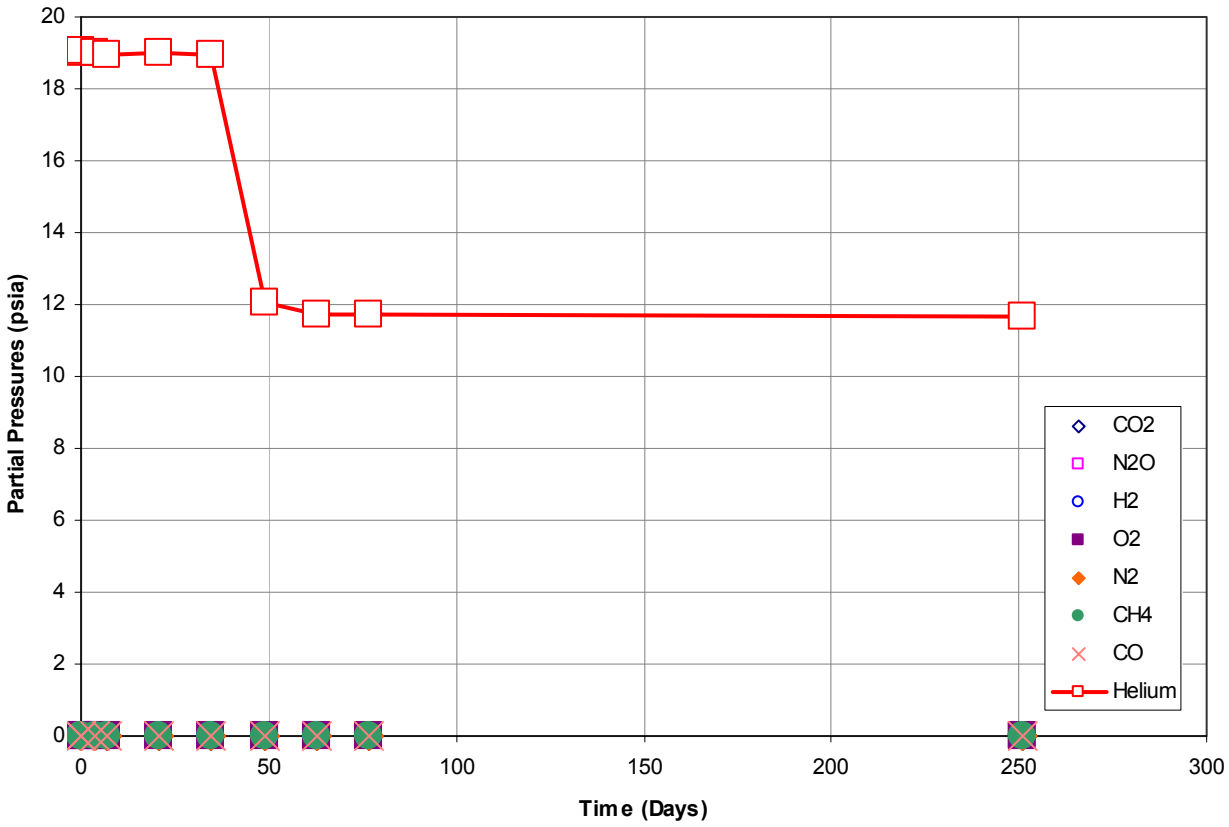
GC Monitoring 3013 Storage Can 1

Can 1, sintered at 950°C and back filled with Helium.

Pressure lowered at 50 days.

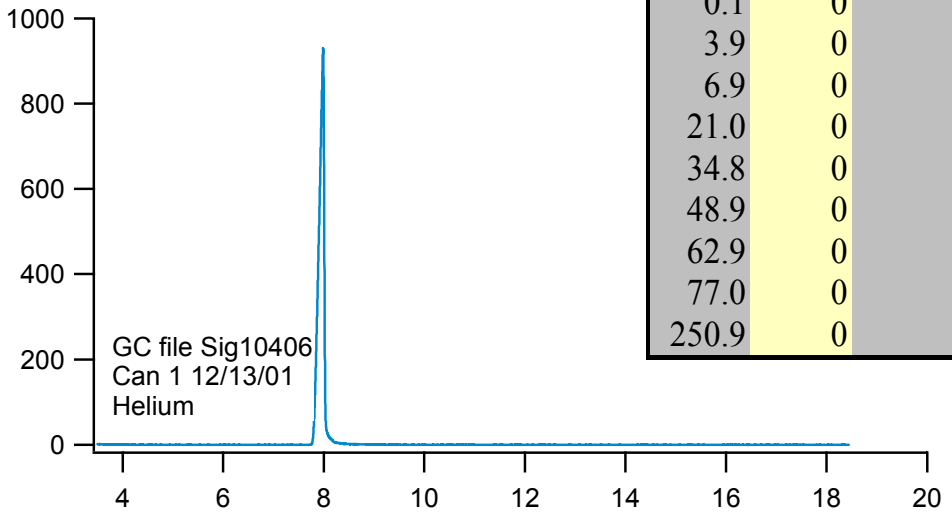
Data are corrected for
- temperature (average of can thermocouples).
- zero point drift of pressure gauge.

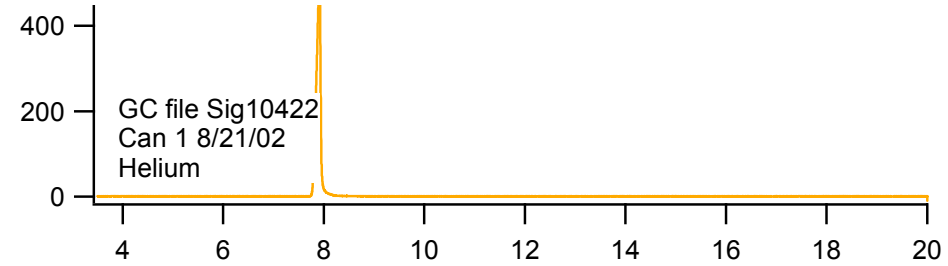
GC analysis is relative and multiplied by measured can pressure.



GC Monitoring 3013 Storage Can 1

Days	CO_2	N_2O	He	H_2	O_2	N_2	CH_4	CO
0.0	0	0	19.9	0	0	0	0	0
0.1	0	0	20.1	0	0	0	0	0
3.9	0	0	20.1	0	0	0	0	0
6.9	0	0	20.1	0	0	0	0	0
21.0	0	0	19.9	0	0	0	0	0
34.8	0	0	19.3	0	0	0	0	0
48.9	0	0	12.9	0	0	0	0	0
62.9	0	0	12.5	0	0	0	0	0
77.0	0	0	12.7	0	0	0	0	0
250.9	0	0	12.5	0	0	0	0	0





Conclusion:

Nothing of consequence in happening in Can 1

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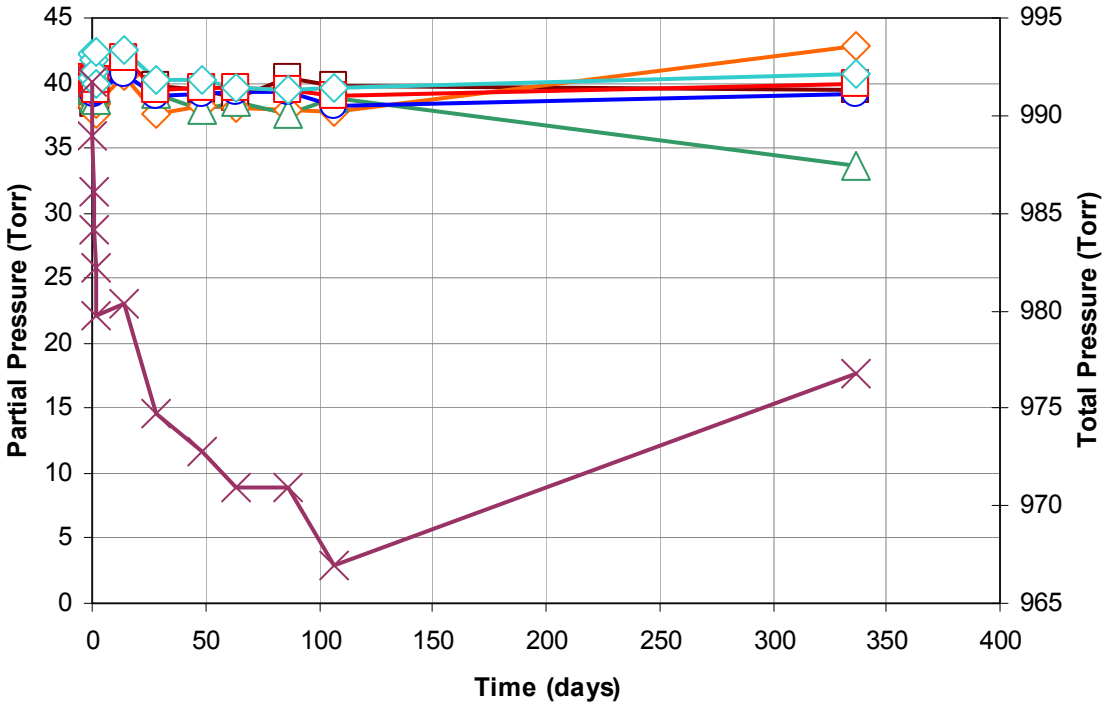
GC Monitoring 3013 Storage Can 10



Purpose of Can 10:

- Reactivity of gases with walls.
- Sample procedure check.

- Pressure rise between 100 and 350 days may be real.
- Change in composition is close to our limits of detection.

Differences in % Composition from Expected

Species	Volume %
CO_2	3.98%
H_2	3.95%
O_2	4.01%
N2	4.03%
CH_4	4.08%
CO	4.07%
Ar	75.88%

Argon is used as the carrier gas and can't be measured, assumed constant.